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IS 1448-120 (1986): Methods of test for petroleum and its products, Part 120: Zinc in libricatinng oil [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS : 1448 [P : 120] - 1986
Adopted : 28 November 1986
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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 120]

ZINC IN LUBRICATING OIL

UDC 621·892 : 543 [546·47]

Adapted with permission from the Institute of Petroleum Standard No. IP 117/82

1. SCOPE

1.1 This method is intended for the determination of zinc in unused lubricating oil. It separates zinc from magnesium and alkaline earth metals, but most heavy metals interfere.

2. SUMMARY OF METHOD

2.1 The sample is ashed and the zinc determined as zinc hydroxyquinolate.

3. REAGENTS

3.1 Sulphuric Acid — Concentrated, relative density 1·84.

3.2 Nitric Acid — Concentrated, relative density 1·42.

3.3 Hydrochloric Acid — 2 N approximate.

3.4 Thymol Blue Indicator — Dissolve 0·1 g thymol blue in 50-ml ethyl alcohol and dilute to 100-ml with water.

3.5 Ammonium Hydroxide — Concetrated, relative density 0·880.

3.6 Oxine Solution — Dissolve 2 g of 8-hydroxyquinoline in 10-ml glacial acetic acid and dilute to 100-ml with water.

3.7 Ammonium Acetate Buffer — Saturated aqueous solution.

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4. PROCEDURE

4.1 Weigh to the nearest 0·01 g into a silica crucible. an amount of sample containing 20 to 40 mg of zinc and heat it gently until the oil ignites. Allow it to burn until a coke residue is formed, maintaining the burning with a gas flame if necessary; and continue heating with a gas flame until all the volatile matter has been driven off. Transfer the crucible to a muffle furnace at 550°C and heat it until the ash is free from all visible traces of carbon.

4.2 Cool, moisten the residue with approximately 2-ml of concetrated sulphuric acid. Heat the crucible until white fumes are evolved and assist the oxidation by the addition of concentrated nitric acid. Heat again until white fumes are evolved.

CAUTION : The concentrated nitric acid shall be added drop-wise and slowly in order to avoid an excessively violent reaction.

4.3 Allow the crucible to cool, add 10 ml of 2 N hydrocloric acid cautiously, and transfer the solution to a 250-ml beaker using approximately 100-ml of water. Heat until the solution just boils and allow it to stand for at least 4 h, or preferably overnight.

4.4 Filter through Whatman No. 42 or similar filter paper, wash with hot water, and add two drops of thymol blue indicator. Add concentrated ammonium hydroxide, drop-wise with stirring, until the solution just changes to blue, then 2 N hydrochloric acid drop-wise until the solution is yellow.

4.5 Heat the solution to 70°C and add 10 ml of oxine solution for each 10-mg of zinc present. Then add 5-ml of amonium acetate buffer solution. Stir the solution, remove from the hot plate and stand for approximately 15 minutes. Filter the precipitate on a weighed No. 3 sintered glass filter (20 to 30 μm porosity). Wash thoroughly with hot water and dry the precipitate to constant mass at 130 to 140°C.

5. CALCULATION AND REPORT

5.1 Calculate the percentage of zinc in the sample from the equation :

$$\text{Zinc, percent by mass} = \frac{18{\cdot}49\, M_1}{M_2}$$

where

M_1 = mass in g of precipitate, and

M_2 = mass in g of sample.

5.2 Report the result to the nearest 0·001 as zinc content, percent by mass.

6. PRECISION

6.1 Results of duplicate test shall not differ by more than the following amounts :

Mean	*Repeatability*	*Reproducibility*
X	$0{\cdot}036\ (x + 0{\cdot}025)$	$0{\cdot}033\sqrt{X}$
0·02	0·001	0·004
0·05	0·002	0·007
0·1	0·004	0·010
0·2	0·008	0·014

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